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MODIFICATION TO MAKE SAGO FLOUR CLOSE TO THE EXTENDER OF UREA FORMALDEHYDE ADHESIVE PROPERTIES *)

(*Modifikasi agar sifat sagu sesuai dengan sifat Ekstender perekat urea-formaldehida*)

By / *Oleh*

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Ringkasan

Sejak tahun 1970, baik jumlah maupun kapasitas pabrik kayu lapis di Indonesia meningkat dengan pesat. Pada tahun 1995, produksi kayu lapis Indonesia mencapai sekitar 13,6 juta meter kubik. Untuk memenuhi produksi sebesar itu diimpor sekitar 240.930 ton tepung gandum. Dapat diduga bahwa tepung gandum dapat diganti oleh bahan pati lain seperti tepung sagu. Tepung sagu seperti tepung gandum, mengandung kadar pati yang tinggi. Karena itu tepung sagu berpotensi tinggi untuk mengganti tepung terigu sebagai bahan ekstender perekat urea-formaldehida (UF).Tetapi perbedaan besar molekul dan kandungan bahan kimia lain seperti gluten menyebabkan sifat fisiko-kimia sagu berbeda dengan terigu bila digunakan sebagai bahan ekstender perekat UF.

Tepung sagu adalah hasil ekstrak batang pohon sagu (Metroxylon sagus Roxb.*). Pohon sagu terutama tumbuh di Indonesia dan Papua Nugini. Selain itu, dalam jumlah kecil juga tumbuh di Malaysia, Thailand dan Philipina. Dari potensi pohon yang tersedia diperkirakan bahwa setiap tahun di Indonesia tersedia sebanyak 3,6 juta ton tepung sagu. Bila tidak dipanen tepung sagu tersebut akan terbuang bersama pohon yang segera mati setelah pembuahan.*

Dalam penelitian ini sifat fisiko kimia tepung sagu yang dimodifikasi diuji dan diharapkan memiliki sifat seperti tepung gandum sebagai bahan ekstender perekat UF. Tepung sagu dimodifikasi dengan dua cara yaitu dengan penambahan asam klorida (HCl) dan dengan fosforilasi (campuran mono- dan di-sodium fosfat) sampai mencapai padanan sifat bahan ekstender perekat UF yang lazim digunakan.

Kata kunci : sagu modifikasi, fosfat modifikasi, sifat fisiko-kimia, sifat ekstender

Summary

Since 1970, both the number and the capacity of Indonesian plywood factories have increased very rapidly. In 1995, Indonesian plywood factories produced about 13.6 million cubic meters of plywood. This production consumed about 240.938 tons of wheat flour that is imported from other countries. It has been proposed that wheat flour can perhaps be replaced by another starchy material. Like wheat flour, sago flour has a high starch content; therefore, it could be a potential substitute for wheat flour as an extender of UF (urea-formaldehyde) adhesives. However, the differences in the molecular size and starch content, like gluten, make sago's physical properties unlike wheat flour when it is used to extend UF adhesives.

Sago flour is water-extracted from the pith of sago (Metroxylon sagus *Roxb.*) *trees. Sago palm trees mainly grow in Indonesia in addition to Papua New Guinea, Malaysia, Thailand,*

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and the Philippines. It is estimated that in Indonesia there are about 3.6 million tons resources of dry sago flour from the sago trees. If there are no harvesting, the tree will die and the sago flour resources will disappear.

In this research modified sago flour was tested for its effectiveness as an extender of UF adhesives. Sago flour was modified by two methods. First by acid modified (by HCl), and the second by phosphorylation (by a mixture of mono- and di-sodium phosphate) to achieve appropriate properties as an extender of UF-adhesives.

Keywords : modified sago, phosphorylated sago, physico-chemical properties, extender properties.

I. INTRODUCTION

By 1995 the real production of wood-based panel factories in Indonesia had reached about 13.6 million cubic meters of plywood. The main product has been interior or type II plywood (Anonymous, 1996). Urea-formaldehyde (UF) is the adhesive of choice for interior plywood. The UF resin is blended with water, hardener and extender to formulate it into an adhesive mix prior to being spread to the veneer surface.

The importance of adding the extender is to control viscosity, to prevent glue line dry-out during panel assembly, and to reduce adhesive cost. Consequently adhesive application (Wheatley, 1991 and Klein, 1980), prepressing and penetration are well controlled. The extenders also possibly improve assembly time and bonding quality of the mixed adhesive (Klein,1980) and prevent dry-out and undercure (Robertson & Wheatley, 1988). As more extender is added to the adhesive mixture, more water also needs to be added to achieve an optimum viscosity with less UF content. A decrease of UF content makes the adhesive more economical, however, the bonding strength of the adhesive is decreased.

Depending on the specific application method, different adhesive viscosities are used. Most Indonesian plywood industries use a speader (Sumadiwangsa,1992) and a viscosity from 1300 to1800 centi Poise. Adhesive viscosity will also be adjusted for the species of wood and temperature of the work site. Viscosity adjustments can be achieved by regulating the amount of extender and water added to the adhesive formulation. Typically one kilogram of resin needs approximately 200 grams of extender, 100 grams of water, and 5 grams of hardener. Generally, every cubic meter of plywood needs about 84 kgs of UF-adhesive and 16.8 kgs of extender. So that, in 1995 for 13.6 million tons plywood production, Indonesia needs 240 938 tons of extender.

The plywood factories in Indonesia use industrial wheat flour as an extender, that is imported from other countries (Sumadiwangsa, 1985). It has been proposed that wheat flour can perhaps be replaced by another starchy material such as sago flour.

Like wheat flour, sago flour has a high starch content; therefore, it could be a potential substitute for wheat flour as an extender of UF-adhesive. However, the differences in the molecular size and starch content, like gluten, make sago's physical properties unlike wheat flour if it is used to extend UF-adhesives.

Indonesia has about a 1,114,000 ha of sago palm trees; 1,000,000 ha were estimated from the wild area, and 114,000 ha were estimated from the cultivated area (Flach, 1983). If there are 16 to 20 trunks harvested in one ha, and each trunk

contains about 850 kgs of pith or 250 kgs of dry flour (Flach, 1984), so that anually Indonesia is able to produce about 4.5 million tons of dry sago flour from the wild stand. This amount does not include the product from cultivated stands.

While this field is still new, some research has been done using sago flour as an extender of UF-adhesive. Sumadiwangsa (1985) used sago flour exclusively and compared it with wheat flour. Then, Sumadiwangsa (1986) examined the use of sago flour mixed with wheat flour. These experiments on sago flour revealed a poorer quality in viscosity than that obtained using only wheat flour. Increasing the viscosity of sago flour probably can be done by modifying sago flour or by adding an enrichment of a substitute prior to using it as an extender. If, the sago flour can be used as an extender of UF-adhesive, there will be significant benefits to Indonesia. It would increase work opportunities, land values, native incomes, and in general the spheres of activity would be increased. Indonesian import of wheat flour could then be reduced. In general the dependence on foreign imports will decrease and the values of Indonesian products will increase.

The applied research on sago flour needs to be continued in order to get the most beneficial utilization of the sago flour. The use of sago flour as an extender for UF-adhesive should be not only practical but also economical. In this research modified sago flour will be tested by physicochemical properties, especially viscosity, for its effectiveness as an extender of UF-adhesives. Also, it will be determined in this research if sago flour (modified and unmodified) can be used in a UF-adhesive formulation to make satisfactory adhesive bonds.

In these experiments sago starch was modified by two methods. First, by soaking the sago sample for seven days in 7.5% hydrochloric acid reagent. In the second modifications the sago flour was soaked one day in the same solution above and then phosphorylated by an experimentally derived mixture of monosodium phosphate and disodium phosphate to achieve appropriate properties as an extender of UF-adhesive as previously mentioned.

II. PURPOSE

The purpose of the research is to modify sago starch to get the properties acceptable as an extender of a UF-adhesive.

III. EXPERIMENTAL DESIGN

A. Material

Sago was obtained from a small sago factory at Bogor, Indonesia.

Urea-formaldehyde glue, catalyst, and extender were received from Georgia Pacific, Resin, Inc. Albany, Oregon, USA. Resin has 60% resin solids.

Chemicals were American Chemical Society reagent grade.

B. Characterization of Samples

Samples to be characterized were:

* Sago unmodified

* Leading extender (wheat flour)
* Sago modified
  - Acid modified
  - Phosphorylated
  - Acid and phosphor modified

C. Procedures

The procedures to characterize sago properties were adopted from Smith (1967)
Moisture content (oven method)
Bulk Density (graduated cylinder)
Hot Paste Viscosity (Brookfield Viscometer)
pH
Precipitation Time (graduated cylinder)
Water Intake (graduated cylinder)

D. SagoModification

Two methods of sago modification were employed in this experiment :

1. Acid modified sago

Sago flour was soaked in 7.5 % HCl at ambient temperature. After one day, the sago flour was neutralized, washed, and dried at room temperature. After drying, the sago properties were characterized. In addition to one day of soaking, there were also 3, 5, 7, and 9 days of soaking treatments.

The most suitable one of the five treatments was selected as the material for the next phosphorylation step. The seven day treatment (Lintner starch, if using potato instead of sago flour) was tested and characterized as acid modified sago from here on termed ("A-sago").

2. Phosphorylated sago

Before phosphorylation, sago flour was treated by one day soaking in 7.5 % hydrochloric acid (HCl) at room temperature.The phosphorylation was carried out using mixture of monosodium hydrogen phosphate (NaH_2PO_4) and disodium hydrogen phosphate (Na_2HPO_4) at a ratio of one to one. The total phosphate salts added was 0.9 %. Five percent of urea was also added to the mixture (percentages are calculated by the weight of air dry sago flour).

Na_2HPO_4 and NaH_2PO_4 each was weighed to about 0.45 grams and also weighed 5.0 grams of urea. All of these reagents were dissolved in 100 grams of distilled water. The mixture was poured into 100 grams of sago flour, mixed well and let stand for 30 minutes, while occasionally being stirred. The mixture was filtered through filter paper (VWR grade 613) using a vacum pump, and then oven dried for 30 minutes. The mixture was phosphorylated in an oven at 140^0 C for two hours. The crude phosphorylated sago, after reaching room temperature, was washed with distilled water and then air dried to obtain the phosphorylated sago from here on termed ("P-sago").

E. Viscosity of Adhesive Mixtures

The adhesive manufacturer proposed the formula for the glue mixture proportions, resin, catalyst, extender, and water were 1070, 75, 460, and 600 lbs, respectively. Even though, in this experiment, the amount of tested extenders (natural, acid modified, and phosphorylated sago), to get certain viscosity was varied, instead of using a fixed quantity 0f 93.5 percent (based on resin solids). In the experiments, the amount of resin, catalyst, water and wheat flour (or tried extender) were reduced to 6.4, 0.4, 2.3 and 3.0 grams, respectively.

IV. RESULTS AND DISCUSSIONS

A. Properties of Natural Sago and Wheat Flour Extender

Almost all of the properties of the sago starch and wheat flour extender that have been tested differ, especially in viscosity, water intake, pH and retrogradation (on time) (Table 1). These four properties are needed to examine due to the role of each plays adhesive performance.

Control of viscosity is the most important function of an extender. Klein (1980) said that an extender should be able to control viscosity as well as to reduce UF-resin consumption. In practical terms, that means the viscosity of an extender needs to be higher than UF resin itself or than the final glue mixture. The hot paste viscosity of four percent natural sago (6533 cPs) is greatly higher than that the wheat flour (8.6 cPs) (Table 1). Knight (1969) previously noted that sago flour was larger in molecular size, and that it has a higher granular size and viscosity compared to other starchy materials.

Table 1. Comparison Between Sago Starch and Wheat Flour Properties
Tabel 1. Perbandingan sifat tepung sagu dan terigu

Properties (*sifat*)	Sago flour (*tepung sagu*)	Wheat flour (*tepung terigu*)
Moisture contents (*kadar air*) , %	14.65	12.06
pH	4.58	5.97
Retrogradation, (*pengenapan*)		
- minutes (*menit*)	31.3	232.3
- % volume (*isi*)	52.1	79.3
Weight/volume, (*berat/isi*)		
- loose (*rambang*), g.cm^{-3}	0.675	0.429
- dense (*tumpat*), g.cm^{-3}	0.856	0.583
Viscosity (*kekentalan*), cPs	6533	8.6
Water intake (daya isap air), g/g, %	160.3	231.2

Remarks (*keterangan*) : Average of 3 replications (*rata-rata dari 3 kali ulangan*)

Water intake is the second important property to examine in the role of extender substitutions. An extender has to be a hydrophilic material, since UF-resins are water borne adhesives. The water intake of natural sago is 160.3 percent, while that of

wheat flour is 231.2 percent. The water intake difference is due to differences in the molecular size of sago compared to wheat flour. In modifying sago flour to have a higher water intake, one method could be to reduce its molecular size. One method to do this is by acid catalyzed cleavage of the inter-glucosyl acetal linkages. Such an acid treatment would have to be carefully controlled to achieve the desired reduction in molecular size and does not complete hydrolysis up to monomer units (glucose).

Retrogradation or precipitation time is another measure of an extenders' property. It is essential that an extender remains suspended within the adhesive. It is essential that an extender remains suspended within the adhesive. If an extender settles (retrogrades) too fast, the glue mix will become unhomogenous and excess water in spots will cause problems with adhesion. In a practical sense, such spots of water in plywood adhesion are a leading cause of panel blows. Thus, localized spots of excess moisture need to be avoided. In addition, at all costs, such localized excess water will also cause over penetration of the adhesive and reduce the adhesive cure rate and thus leading what is (Karchesy, 1994) referred to as " glue line wash out" in the plywood industry. This situation also needs to be avoided at all costs. Natural sago has a faster retrogradation time (31.3 minutes) compared to wheat flour (232.2 minutes) (Table 1).

B. Acid Modification of Sago

The aim of acid modification is to make the sago flour a fluid mixture which is less viscose than that the mixture using sago in its original state. Viscosity of acid modified sago depends on the acid concentration used to modify the starch, reaction time, temperature of the modification reaction (Rohwer and Klem, 1984 and Wurzburg, 1986), as well as the type of acid used (Wurzburg, 1986). In this experiment 7.5 % HCl solution was used as a modification reagent. It has been shown that acid modified starch does not change the physical form of starch, insolubility in cold water, and birefringence properties according to Shildneck and Smith, 1967. However, less drastic but subtle changes do occur. Compared to the parent starch, acid modified starch has a) lower hot paste viscosity, b) a higher alkali number, c) higher ratio of cold to hot paste viscosity, d) higher osmotic pressure, e) higher gelatinization temperature, f) higher critical absorption of sodium hydroxide, g) lower intrinsic viscosity, h) lower iodine affinity, and i) less granule swelling during gelatinization (Shildneck and Smith, 1967).

Acid modification experiments done on sago flour indicated that viscosity and water intake were successfully altered (Table 2). Both properties near those of wheat flour were obtained. After one day of soaking in 7.5 % HCl, sago has the viscosity (6.9 cPs) which is near that of wheat flour (8.6 cPs). After more than one day of soaking the viscosity persistently decreases, and after nine days of soaking the viscosity is 2.1 cPs that is very low (Table 2).

The longer the acid soaking, the greater the water intake in acid modified sago. After seven and nine day periods of soaking the water intake of acid modified sago (203.5 and 202.8 g/g) is near the same level of wheat flour at 231.2 g/g (Table 2).

After seven and nine day periods of soaking, the retrogradation and the properties seemed to remain constant as they did with natural sago (Table 2).

Table 2. Sago Starch Properties Affected by Various Days of Soaking in 7.5 % HCl Acid

Tabel 2. Sifat tepung sagu setelah beberapa hari direndam dalam 7,5 % asam klorida

Properties (*sifat*)	HCl soaking period (*lama perendaman*), days(*hari*)					Wheat Flour (*tepung terigu*)
	0	1	3	7	9	
Moisture content (*kadar air*), %	14.6	14.3	13.7	14.3	13.3	12.1
pH	4.58	4.93	4.93	4.89	5.01	5.97
Retrogradation (*Pengenapan*)						
- minutes, (*menit*)	31.3	36.3	36.7	38.3	39.0	232.3
- % volume, (*isi*)	52.1	53.0	57.2	59.0	59.4	79.3
Weight/volume (*berat/isi*)						
- Loose (*rambang*)	.675	.711	.695	.692	.677	.429
- Dense (*tumpat*)	.856	.861	.826	.865	.848	.583
- Viscosity (*kekentalan*), cPs	6533	6.9	3.1	2.3	2.1	8.6
- Water intake (*daya isap air*), %	160.3	162.7	182.1	203.5	202.8	231.2

Remarks (*keterangan*) : Average of 3 replications (*rata-rata dari 3 ulangan*)

C. Phosphorylated Sago

Phosphorylated of natural sago increased its pH, retrogradation, water intake and viscosity (> 10,000 cPs) as can be seen in Table 3. These results are consistent and similar to those reported by Kerr (1960). Since the viscosity of phosphorylated sago (> 10,000 cPs) was unusable in this range, it was decided to explore phosphorylation effects on one day acid treated sago. Table 3 indicates that this combination of treatments gave material with an acceptable viscosity value of 13.0 cPs. In fact, all of the values in Table 3 for the phosphorylated-acid soaked sago, compare very favorably to those of the wheat flour standard also shown in Table 3. The retrogradation value of 122 minutes is about half the time length time for wheat flour (232 minutes), but this length of time is judged to be an adequate time for any adhesive mixture assembly time.

The influence of varying amounts of water on viscosity with selected tested extenders is shown at Figure 1 for natural sago (N-sago), Figure 2 acid modified sago (A-sago), and Figure 3 phosphorylated sago (P-sago). As one would expect, increasing water content decreases viscosity as exhibited by these figures. The regressions are exponential with the equations as follows :

Sago samples	% Solid resin weight	Equations
Natural sago-A	94	Y = Exp (-0.028 x) * 1808.3
Natural sago-B	130	Y = Exp (-0.054 x) * 6939.8
Natural sago-C	182	Y = Exp (-0.051 x) * 14588.9
Acid modified	52	Y = Exp (-0.062 x) * 2299.6
Acid modified	78	Y = Exp (-0.046 x) * 4156.5
Acid modified	130	Y = Exp (-0.039 x) * 7764.7
Phosphorylated	38	Y = Exp (-0.061 x) * 2733.0
Phosphorylated	78	Y = Exp (-0.045 x) * 5693.2
Phosphorylated	130	Y = Exp (-0.040 x) * 17574.3
Without	0	Y = Exp (-0.036 x) * 377.7

Table 3. Phosphorylated Sago Properties from Natural and One Day Acid Soaking

Tabel 3. Sifat sagu hasil fosforilasi dari sagu alami dan sagu asam

Properties (*sifat*)	Sago natural (*sagu alami*)	Phosphorylated sago		Wheat flour (*tepung terigu*)
		natural (*alami*)	acid soaks (*rendam asam*)	
Moisture content (*kadar air*), %	14.65	13.35	13.15	12.06
pH	4.58	6.87	5.85	5.97
Retrogradation (*Pengenapan*)				
- minutes, (*menit*)	31.3	71.3	121.7	232.3
- % volume, (*isi*)	52.1	63.4	74.2	79.3
Weight/volume (*berat/isi*)				
- Loose (*rambang*)	.675	.610	.660	.429
- Dense (*tumpat*)	.856	.811	.772	.583
- Viscosity (*kekentalan*), cPs	6533	> 10000	13.0	8.6
- Water intake (*daya isap air*), %	160.3	192.3	224.7	231.2

Remarks (*keterangan*) : Average of 3 replications (*rata-rata dari 3 ulangan*)

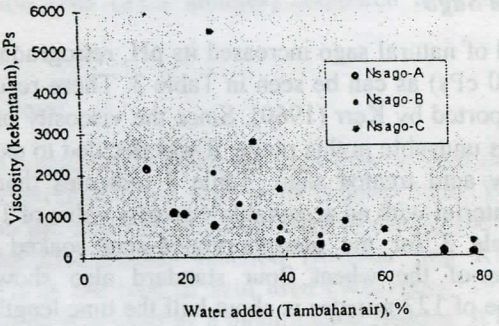

Figure 1. The effect of proportions of natural sago and water on viscosity of adhesive mixtures.

Gambar 1. Pengaruh proporsi sagu alami dan air terhadap kekentalan campuran perekat.

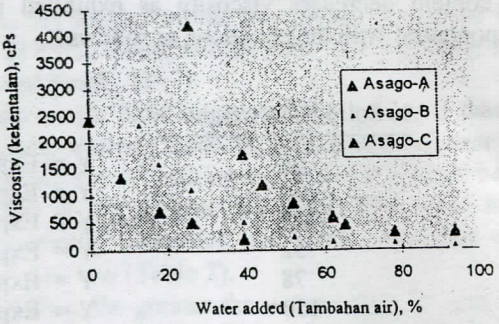

Figure 2. The effect of proportions of acid-modified sago and water on viscosity of adhesive mixtures.

Gambar 2. Pengaruh proporsi sagu asam dan air terhadap kekentalan campuran perekat.

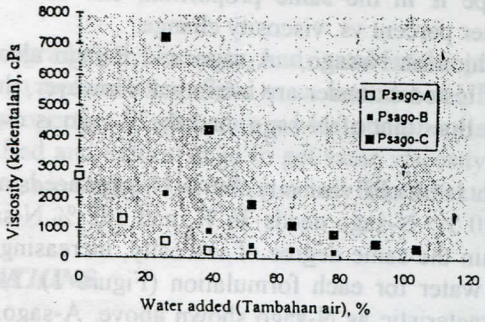

Figure 3. **The effect of proportions of phosphorylated sago and water on viscosity of adhesive mixtures.**

Gambar 3. ***Pengaruh proporsi sagu fosforilasi dan air terhadap kekentalan campuran perekat.***

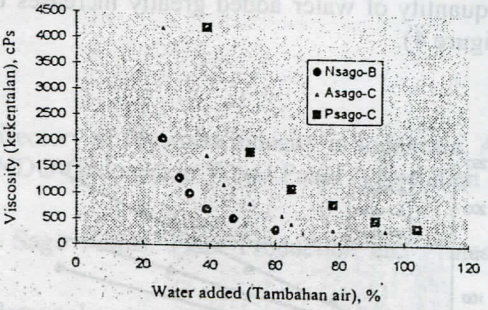

Figure 4. **The effect of proportions of modified sago and water on viscosity of adhesive mixtures.**

Gambar 4. ***Pengaruh proporsi sagu modifikasi dan air terhadap kekentalan campuran perekat.***

From Figures 1, 2, and 3 one can see the similar curves for viscosity vs. amount of water added to each tested extender mix. Figure 1 shows the curves for the natural sago/adhesive mixtures N-sago-A, N-sago-B and N-sago-C.

Given the same amount of added water, N-sago-A has the lowest viscosity while N-sago-C has the highest. N-sago-B is intermediate. For example, from Figure 1 one can see that at 50 % added water to the adhesive mix, N-sago-C gave a viscosity of 230 cPs while N-sago-B gave 470 cPs and N-sago-A had a viscosity of 900 cPs.

In Figure 2, A-sago-A has the ratio 0.52 to 1.00 of acid modified sago to adhesive, A-sago-B 0.78 to 1.00 and A-sago-C 1.30 to 1.00. In Figure 3 the ratio of phosphate modified sago to adhesives mix is 0.38 to 1.00 for P-sago-A, 0.78 to 1.00 for P-sago-B and 1.30 to 1.00 for P-sago-C.

Figure 4 compares the relative change in viscosity with increasing percentages of water for natural, acid treated and phosphorylated sago extenders, where each extender type is in the same proportion in the adhesive mixture (1.3 to 1.0 extender to U-F adhesive). These are designated as N-sago-B, A-sago-C, and P-sago-C. Since

each sago flour type is in the same proportion, one can compare the effects o modification on water percent vs. viscosity change.

In the same fashion as N-sago, A-sago and P-sago also increase in viscosity when the amount of tested extender are increased. However, the increase of viscosity of A-sago is higher than that of N-sago, and the P-sago is even higher as shown a Figure 4.

In order to reach 1000 cPs viscosity, 94 % N-sago needs a total amount of wate of about 21 % ; 130 % N-sago needs 36 %, and 182 % N-sago needs 52 % wate (Figure 1). To obtain the same degree of viscosity, increasing percentage of N-sag needs more added water for each formulation (Figure 1). A-sago and also P-sag have the same characteristic as N-sago shown above. A-sago, however, needs mor water than N-sago, and P-sago even more water than A-sago to get the sam viscosity at the same proportions of materials (Figure 4). These results suggest tha for the purpose of extending a urea-formaldehyde resin, A-sago is better than N-sago but P-sago is the best.

To obtain a viscosity of more than 1000 cPs, the effect of water content is mor sensitive. A small quantity of water added greatly increases the viscosity of all th tested extenders (Figure 4).

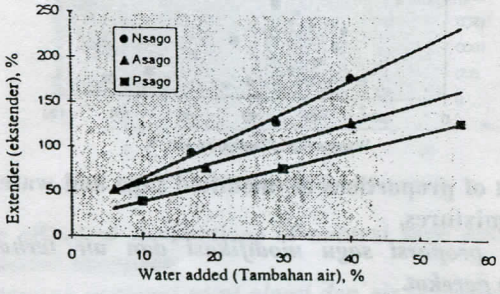

Figure 5. **The correlation between tested extenders and proportion of water i adhesive mixtures to get 1500 cPs viscosity**

Gambar 5. ***Hubungan antara ekstender yang dicobakan dengan berat air yan ditambahkan pada campuran perekat untuk mendapatkan kekentala 1500 cPs***

At 100 % N-sago in glue mixture, to reach a viscosity of 1500 cPs need a tota water about 19 %, A-sago needs 28 %, and P-sago needs 41 % water (Figure 5). A 130 % N-sago, A-sago, P-sago to reach 1500 cPs on viscosity needs 28, 42, and 6 % water, respectively. To get the same viscosity at the same amount of teste extender, A-sago needs water about 1.5 times of N-sago, and P-sago needs twice a much water as N-sago. Again for the purpose of extension A-sago is better than N sago, and P-sago is the best. The amount of water needed in the adhesive mixture blended with P- sago is very similar to that required by industrial wheat flour which is now used in Indonesia (Sumadiwangsa, 1985).

V. CONCLUSIONS

Based on pH, retrogradation, viscosity, and water intake properties, phosphorylated sago acceptable to replace wheat flour as an extender of UF-adhesive.

Again, from the properties of glue mix where phosphorylated sago need more water than acid modified and natural sago to get same viscosity it can be concluded that phosphorylated sago is the best to replace wheat flour to extend a UF-adhesive.

VI. RECOMMENDATIONS

Recommendations for future research are that the formulations developed in this research be tested to make laboratory plywood production prior to actual plywood specimens. Adjusments may have to be made in any scale-up process, but the principles outlined should hold up in the actual production of plywood using urea-formaldehyde extended by phosphorylated sago starch products.

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